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Effect of CAD/CAM aesthetic material thickness and translucency on the polymerisation of light- and dual-cured resin cements

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ABSTRACT

Objectives: This study investigated potential variations in polymerisation of light- and dual-cured (LC and DC) resin cements photoactivated through four CAD/CAM restorative materials as a function of substrate thickness.

Methods: Four CAD/CAM materials [two resin composites CeraSmart (CS) and Grandio Blocs (GB); a polymer infiltrated ceramic Vita Enamic (VE) and a feldspathic ceramic Vita Mark II (VM)], with five thicknesses (0.5, 1, 1.5, 2, and 2.5 mm) were prepared and their optical characteristics measured. 1 mm discs of LC and DC resin cement (Variolink® Esthetic, Ivoclar AG) were photoactivated through each specimen thickness. After 1 h post-cure, polymerisation efficiency was determined by degree of conversion (DC%) and Martens hardness (H_M). Interactions between materials, thicknesses and properties were analysed by linear regressions, two-way ANOVA and one-way ANOVA followed by *post hoc* multiple comparisons ($\alpha = 0.05$).

Results: All substrates of 0.5- and 1.0-mm thickness transmitted sufficiently high peak irradiances at around 455 nm: (I_t = 588–819 mW/cm^2) with translucency parameter TP = 21.14 – 10.7; ranked: CS > GB = VM > VE. However, increasing the substrate thickness (1.5–2.5 mm) reduced energy delivery to the luting cements (4 – 2.8 J/cm^2). Consequently, as their thicknesses increased beyond 1.5 mm, H_M of the cement discs differed significantly between the substrates. But there were only slight reduction of DC% in LC cements and DC cement discs were not affected.

Significance: Photoactivating light-cured Ivocerin™ containing cement through feldspathic ceramics and polymer-infiltrated ceramics achieved greater early hardness results

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than dual-cured type, irrespective of substrate thickness (0.5 – 2.5 mm). However, only 0.5 and 1 mm-thick resin composites showed similar outcome (LC > DC). Therefore, for cases requiring early hardness development, appropriate cement selection for each substrate material is recommended.

1. Introduction

Monolithic digitally designed and fabricated materials are now available that mimic the translucency of natural tooth structure [1]. In addition to their improved biocompatibility [2] and reparability [3], resin-based CAD/CAM blocks revealed distinctively superior optical and mechanical qualities at minimal thickness of 0.3–0.5 mm [4,5]. However, the long-term clinical success of any aesthetic indirect restoration is mainly reliant on adhesive bonding to tooth structure and durability of the natural colour blend [6]. Obtaining sufficient polymerisation of the adhesive resin cement is the first stage in assuring these qualities for restoration longevity [6,7].

Light-cured resin cements (LC) are advocated as the primary choice for aesthetic restorations thinner than 1.5 mm [8,9]. This recommendation is primarily based on their enhanced shade matching and long-lasting colour stability beneath highly translucent restorations, as compared to the yellowing effects associated with dual- and self-cured varieties [6,10,11]. However, thicker aesthetic restorations (2–3 mm) require the use of dual-cured (DC) resin cements [12,13]. Although DC luting cements often had greater mechanical properties and *degree of conversion* (DC%) compared to the LC [14,15], this was not always true for cases with limited light transmission [15–20]. Unlike self-cured luting cements, LC and DC require sufficient light energy, of appropriate wavelength range, for effective monomer conversion [8], described as minimum energy requirement (MER) [21].

Several internal and external factors influence the polymerisation of these adhesive resin cements. External variables such as the light curing unit (LCU) [12,22], the restorative substrate [23,24], the ageing time and condition after curing were investigated [25]. As regards the LCU, polymerisation of resin cements could be affected by its wavelength distribution [26], the radiant exposure received [27], the duration of exposure [24], and the tip diameter and its distance from the restoration [26,28]. Different instruments such as reflective spectrophotometers and MARC™ systems have been used to characterise the light transmitted through different substrates [21,29,30]. Variations in interrelated optical properties - translucency, opacity, absorbance, reflectance and light scattering - define the appearance of restorative materials [30,31]. Furthermore, these optical features regulate the light irradiance that penetrates to the underlying luting cement, required to initiate monomer conversion [1,23]. These light attenuating features depend upon the substrate material's microstructural composition, shade and thickness [18,32–34].

The quality of the polymerised cement is also influenced by its intrinsic characteristics, such as: filler particles, polymer matrix, initiator composition and concentration [11,35–37]. A new amine-free photoinitiator with improved lighter shades and discolouration resistance, Ivocerin™ (Ivoclar Vivadent), has been introduced for their light- and dual-cured aesthetic resin cements. Some recent investigations found these Ivocerin™ containing cements to be favourable in terms of degree of conversion, flexural strength and bond strength to dentin [7,38], but comparable to conventional cements with regard to water sorption and colour stability [11].

The polymerisation efficiency of resin composites has been determined using direct and indirect methods in dynamic or static measurements [29]. Fourier transform infrared (FTIR) spectrometer with an *attenuated total reflection* (ATR) accessory is the most employed analytical method to quantify DC% for dental resin composites [29,39,40]. However, adequate polymerisation could be estimated indirectly by monitoring the development of optical or mechanical properties associated with the polymerisation process [29]. Changes in refractive index or microhardness (Vickers and Knoop) have been used to indicate the progress of polymerisation [32,33,41]. Recently, Martens hardness (H_M) has been measured *via* a series of force-controlled indentations to analyse the ageing behaviour of six dual-cured luting cements after photoactivation through a 1-mm thick zirconia substrate over 7 d [25]. Results indicated that complete polymerisation was achieved after 2 d post curing.

The effect of ceramic substrates including composition, shade and thickness on the polymerisation of different luting cements has been determined using degree of conversion and classical microhardness measures [1,7,32,38]. However, conflicting polymerisation results were noted for dual cured luting cements underneath CAD/CAM resin-based substrates compared to ceramic substrates [23,33,40].

Fairly rapid attainment of adequate mechanical properties of luting cements is essential for either completion of the patient treatment or for further clinical procedures such as occlusal adjustments, polishing and impression taking. There is a need to investigate the effect of overlying aesthetic CAD/CAM restorative materials on the polymerisation of amine-free resin cements corresponding to the limited time of a clinical session. Therefore, this study investigated the polymerisation efficiency of two versions (LC and DC) of Ivocerin™ – containing resin cement. These were photoactivated with a blue light curing unit through four CAD/CAM aesthetic materials as a function of thickness and optical characteristics. The null hypotheses were:

Table 1 – Materials included in the study and their available information.

Material Type	Code	Brand name and shade	Composition (wt%)	Manufacturer	Lot no.
Resin composite	**CS**	CeraSmart A2 HT	71% silica (20 nm) and Ba glass (300 nm) nanoparticles 29% Bis-MEPP, UDMA, DMA	GC dental products, Europe	1512091
	GB	Grandio blocs A2 HT	86% nanohybrid fillers 14% UDMA, DMA	VOCO GmbH, Germany	2122435
Polymer-infiltrated ceramic network	**VE**	Vita Enamic 2M2-HT	86% feldspar ceramic porous structure 14% UDMA, TEGDMA	VITA Zahnfabrik, Germany	55310
Feldspathic ceramic	**VM**	Vitablocs Mark II 2M2C	Fine-particle feldspar ceramic	VITA Zahnfabrik, Germany	91170
Adhesive resin cements (amine-free)	**LC**	Variolink® Esthetic LC Light	Light-cured resin cement, UDMA, ytterbium trifluoride, Ivocerin (initiator), stabilisers	Ivoclar Vivadent AG, Liechtentein	Z01061
	DC	Variolink® Esthetic DC Light	Dual-cured resin cement, UDMA, ytterbium trifluoride, Ivocerin (initiator), stabilisers	Ivoclar Vivadent AG, Liechtentein	Z017C4

1. The optical properties of the CAD/CAM materials did not vary with thickness.
2. The DC% and H_M of the LC luting cement did not vary with either CAD/CAM materials or their thickness.
3. The DC% and H_M of the DC luting cement did not vary with either CAD/CAM materials or their thickness.

2. Materials and methods

2.1. Study design

Four commercially available aesthetic CAD/CAM materials were investigated (Table1): two resin composites (**CS** and **GB**), a hybrid ceramic (**VE**), and a feldspathic ceramic (**VM II**). A total of 120 specimens were prepared into plate form of five clinically relevant thicknesses of 0.5, 1.0, 1.5, 2.0, and 2.5 mm (n = 6). The study flowchart is illustrated in Fig. 1. The optical properties were measured for each specimen thickness using a spectrophotometer and visible light transmission spectrometry (MARC™-LC). A total of 144 disc-shaped specimens (n = 3 per subgroup) from LC and DC adhesive resin cements (Variolink® Esthetic, Ivoclar Vivadent) were photoactivated through each CAD/CAM specimen thickness and one without any substrate (control). The polymerisation efficiency of the luting resins after 1 h post-curing was determined using two measurements: i) degree of conversion (DC%) using Fourier Transform Infrared Spectroscopy (FTIR), and ii) Martens hardness (H_M) of the top surface.

2.2. Specimen preparation

Specimens (n = 6) from each CAD/CAM block were sectioned into *plates* of five thicknesses (0.5, 1, 1.5, 2 and 2.5 mm) using a water-cooled diamond sectioning saw (IsoMet 1000 Precision saw, Buhler®). Specimen thickness was measured with a digital micrometre (± 0.1 mm). The specimens were polished under running water successively using P600, P800, P1000 grit silicon carbide papers at 350 rpm (MetaServ™ 250 single grinder-polisher, Buhler®, USA). All 120 specimens were ultrasonically cleaned in distilled water for five min,

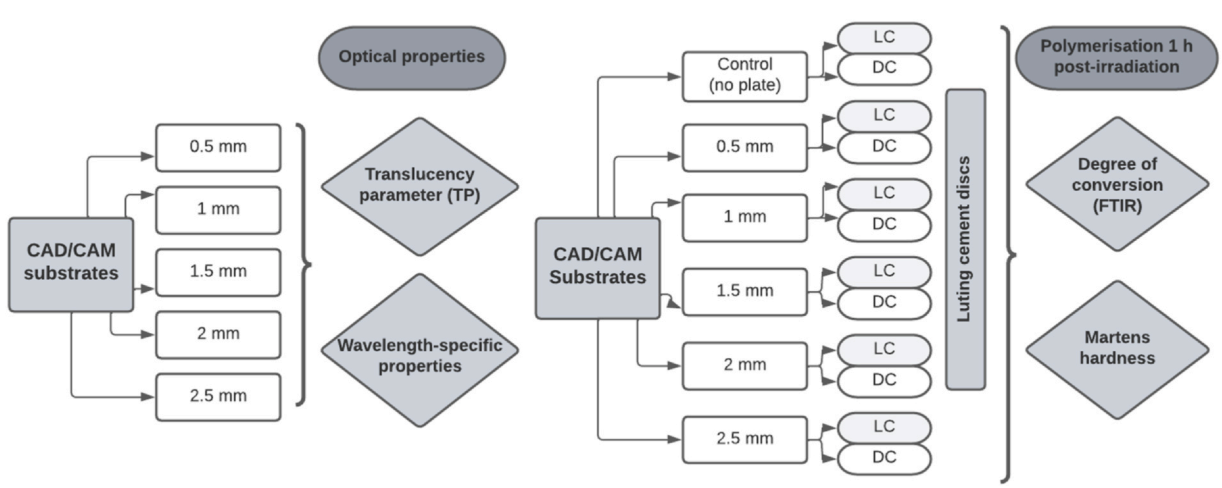

Fig. 1 – Flowchart for production of each CAD/CAM substrate and the light-cured (LC) and dual-cured (DC) luting cement discs.

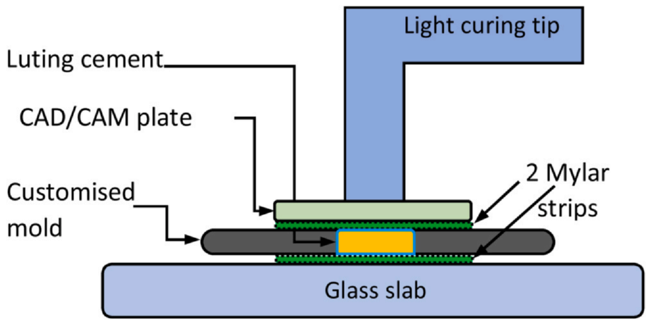

Fig. 2 – Schematic of the luting cement discs cured through CAD/CAM substrates. Representative images for several experimental steps are presented in the supplementary document.

then were stored dry at room temperature in labelled containers.

To standardise dimensions of luting cement specimens, a mould with disc-shaped opening (10 mm diameter and 1 mm thickness) was placed over a glass slab (Fig. 2). Each opening was filled with the luting cement and sandwiched between 0.6 mm Mylar strips on top and base. A standard output LED-LCU (Elipar™ S10, 3 M ESPE, Germany) was used of wavelength range 430–480 nm with the optic tip directly contacting the plate for 20 s. At the start of the experiment, the radiant emittance (1200 mW/cm^2) was verified using a calibrated radiometer (MARC-LC™: Blue-light Analytics Inc, Halifax, Canada). Luting cement discs with defects or air bubbles were discarded.

For each substrate group, six specimens from both luting cement types were photoactivated by transillumination through the CAD/CAM substrates. Control specimens from each cement type were light cured through a glass slide (1 mm thick), without any CAD/CAM plates. No surface treatment nor bonding agent was applied to the CAD/CAM plates. For each cement type, a total of 288 cement specimens were prepared and subdivided into two groups. One group was scanned by FTIR spectroscopy in real time mode up to 60 min. The second group was labelled and stored in dark glass containers containing distilled water at 37 °C for 1 h before instrumented Martens indentation.

2.3. Optical properties of aesthetic CAD/CAM materials

2.3.1. *Translucency parameter (TP)*

Each CAD/CAM specimen (n = 6 *per material thickness*) was placed on the 6 mm-diameter aperture of the reflective spectrophotometer (LabScan XE, HunterLab, USA). For each specimen, four CIE L*a*b* colour coordinates relative to the standard illuminant D_{65} were recorded against a black background (L* = 0.017, a* = 0.015, and b* = 0.001) and four coordinates against a white background (L* = 98.82, a* = 0.065, and b* = 0.123). The same black and white standard tiles were used to calibrate the instrument at the start of each session. TP_{Lab} was determined by calculating the colour difference in the L*, a*, b* measurements for each specimen against white (W) and black (B) backgrounds using Eq. (1):

$$TP_{Lab} = \sqrt{(L_W - L_B)^2 + (a_W - a_B)^2 + (b_W - b_B)^2} \tag{1}$$

A material is completely transparent if TP is 0 and opaque if TP is 100.

2.3.2. *Wavelength-specific optical characteristics*

The peak transmitted light irradiance at around 455 nm (Fig. S5) was recorded for each CAD/CAM specimen thickness (n = 6). Two readings were taken for each specimen using the MARC-LC™ device (Blue-light Analytics Inc, Halifax, Canada). The radiant emittance of the LED light curing unit was 1200 mW/cm^2 with a wavelength range of 430–480 nm and a 9 mm-diameter output (Elipar™ S10, 3 M, Seefeld, Germany). CAD/CAM specimens were centred on the 3.9-mm diameter-sensor of the radiometer. The mean light irradiance was measured in real time by fixing the LCU tip on the CAD/CAM plate and using silicone putty to shield the specimens from ambient light.

The apparent transmission (T′ %), opacity (O_p), and apparent absorbance (A′) were calculated using Eqs. (2)–(4), respectively [42]:

$$T'\% = \frac{I_t}{I_0} \times 100 \tag{2}$$

$$O_p = \frac{I_0}{I_t} \tag{3}$$

$$A' = \log_{10}\left[\frac{1}{T}\right] \tag{4}$$

I_t represents the irradiance of the transmitted light beam and I_0 is the irradiance of the incident light beam. Where T = 100 indicates complete light passage through the material and T = 0 indicates complete light absorption by the material.

2.4. Polymerisation of the luting cements (1 h post-curing)

2.4.1. *Degree of conversion (DC%)*

Degree of conversion (%) was measured in real-time over 1 h by Fourier transform infrared spectroscopy (FTIR, ALPHA II, Bruker, USA) with an attenuated total reflectance (ATR) accessory. The percentage of C=C group conversion (DC%) was measured using the following parameters 4000–400 cm^{-1} wavelength, 4 cm^{-1} resolution, and taking one spectrum per 4 s

A custom 3D-printed mould was stabilised over the ATR crystal with a circular hole of diameter 10 mm and 1 mm depth (N = 144; 3 luting discs × 2 curing types × 4 CAD/CAM materials × 6 thicknesses). The spectra of the uncured luting cement specimens were measured over the first 8 s (2 scans). Then, the luting cement discs (n = 3) with a Mylar strip in between, were irradiated through the corresponding CAD/CAM plate by the LCU for 20 s. FTIR spectra were recorded for 1 h *via* OPUS software (BRUKER OPTIK GmbH, Germany). The DC% calculation used the two-frequency technique for the absorbance peak height ratio where the analytical frequency aliphatic C=C peak at 1618 cm^{-1} was normalised against the (reference frequency) aromatic C=C at 1590 cm^{-1} according to Eq. (5) [27]:

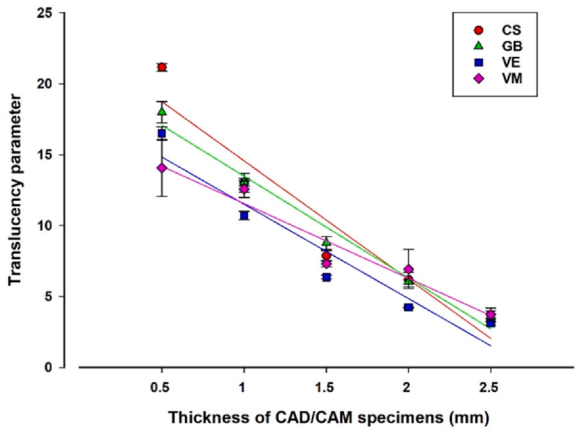

Fig. 3 – Linear regressions of translucency parameter *versus* thickness of CAD/CAM substrates (Pearson correlation, r^2 = 0.91 – 0.98, $p = 0.001$).

$$DC(\%) = \left[1 - \left(\frac{\left(\frac{Aliphatick}{Aromatic}\right) peak\ height\ post - polymerisation}{\left(\frac{Aliphatick}{Aromatic}\right) peak\ height\ pre - polymerisation}\right)\right] \times 100 \quad (5)$$

2.4.2. Martens hardness (H_M)

Each luting disc was removed from distilled water (37 °C) after 1 h and dried lightly with an absorbing paper for 5 min (N = 144). A Martens hardness instrument (Z2.5, ZwickRoell Ltd., Ulm, Germany) was used with a Vickers hardness measurement tip. A fixed distance (12 mm) was maintained between the top surface of the disc and the hardness measuring head at the start of all measurement sessions. A force up to 10 N was applied at a loading speed of 5 N/s, maintained for 30 s and then removed at a rate of 5 N/s. The initial approach rate was 200 mm/min, while the approach speed of the indenter tip until initial contact was 40 mm/min. The sensor tip distance to each specimen after proximity was 40 μm.

Four force-controlled and equally spaced indentations were made on the top surface, in the centre of each disc (n = 3). Martens hardness (H_M) was obtained *via* software (TestXpert®, Zwick GmbH & Co, Ulm, Germany) based on Eq. (6) in ISO-14577–4/2016 [43]:

$$H_M = \frac{F}{A_s\ (h)} = \frac{F}{26.43^*h^2} \quad (6)$$

H_M was expressed in N/mm^2, F is the load in N, $A_s(h)$ is the surface area of the indenter at a distance h from the tip in mm^2.

2.5. Statistical analysis

Data were analysed using statistical software (SPSS 22.0; IBM SPSS Statistics Inc., Chicago, IL, USA). Shapiro-Wilk and Levene's tests were used to confirm the normality and homogeneity of variance, respectively. Two-way ANOVA was used to determine any interaction between the CAD/CAM materials and their thickness for the optical properties. One-way ANOVA, followed by multiple comparison Games-Howell *post hoc* tests (α= 0.05), were conducted to determine any differences in the optical properties between thickness groups within a single material and between the materials within a single thickness. Similarly, the luting resin cements were analysed following the above statistical tests. Dunnett's *post hoc* tests were used for comparing each luting disc to its control (α= 0.05). Paired t-tests were performed to investigate any differences between the two types of luting cements (α= 0.05).

Linear regression models were obtained to analyse the relationships between TP and irradiance (I_t) *versus* thickness of the CAD/CAM materials. Pearson correlation was used to analyse relationships between DC% and H_M and I_t for both luting cements. G'power software (V. 3.1.3; Heinrich Hein University, Germany), *post hoc* power analysis indicated that the luting cement sample size of 144 and CAD/CAM specimens of 120 provided sufficient statistical power to reject the null hypotheses in this study.

3. Results

3.1. Optical properties of aesthetic CAD/CAM materials

3.1.1. Translucency parameter (TP)

Results are presented in Table S1 and the correlations between the TP and specimen thickness are illustrated in Fig. 3.

Strong inverse linear correlations were confirmed between TP of each material and their thicknesses (r^2 = 0.91–0.98, $p = 0.001$). TP were significantly different between the four CAD/CAM materials ($p = 0.0001$).

At 0.5 mm substrate thickness, the average TP increased in the following sequence VM < VE < GB < CS (ranging from 14.2 to 21.1). While at 2.0 and 2.5 mm, TP reduced in all materials (CS = GB = VM), with VE being the least translucent ($p = 0.001$) and significantly different from the other materials.

3.1.2. Optical characteristics at 455 nm

Results are presented in Table S2 and correlations between the CAD/CAM substrates and different optical parameters are illustrated in Fig. 4.

The peak spectral absorption for the luting cements was around 455 nm, (Fig. S5). Therefore all changes in optical properties were tabulated at 455 nm. The mean irradiance (mW/cm^2) at 455 nm decreased linearly with CAD/CAM thickness. For each of the four materials, r^2 was in the range 0.94 – 0.96, ($p = 0.0001$). The apparent transmittance (T′ %) positively correlated with the translucency parameter (TP): r^2 = 0.967, $p = 0.0001$.

The incident irradiance (I_0) was 1200 mW/cm^2. However, after passing through the CAD/CAM substrates, I_t ranged from 182 to 819 mW/cm^2 – depending on each material and its thickness - corresponding to an apparent transmittance (T′ %) of 15–68%.

The transmitted irradiance (I_t) was significantly different between all materials, except for GB and VM at 0.5- and 1-mm thicknesses.

At 0.5 thickness, the measured I_t and T′ % increased in the following sequence, VE < VM = GB < CS.

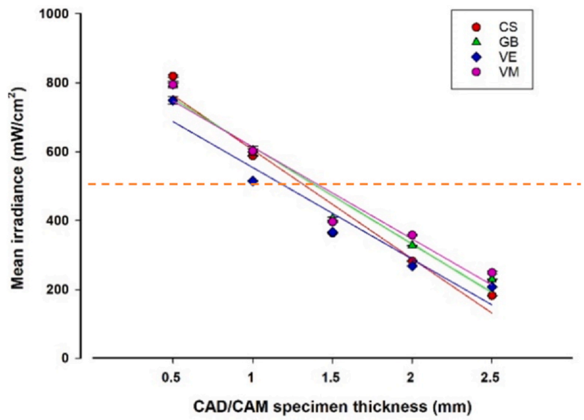

Fig. 4 – Linear regressions of mean irradiance at 455 nm *versus* substrate thicknesses (r^2 = 0.94 – 0.96, p = 0.001). The horizontal dashed line indicates the minimum irradiance (500 mW/cm^2) recommended for polymerising these luting cements.

At 0.5 and 1 mm, all materials had I_t above 500 mW/cm^2 which represent the minimum irradiance recommended for sufficient polymerisation of Ivocerin™-containing luting cements for 20 s [32]. However, this minimum irradiance was not met in substrates thicker than 1 mm. At higher thicknesses (1.5 – 2.5 mm), the irradiance (I_t) passing through all CAD/CAM materials reduced by between 66% and 85%.

At 2- and 2.5-mm thickness, VM substrates had the highest I_t compared to other materials.

3.2. *Polymerisation of the luting cements*

3.2.1. *Degree of conversion (DC%)*

The mean and standard deviation of DC% for luting cements (LC and DC) after 1 h post-curing through each CAD/CAM material are presented in Table S3 and graphically illustrated in Fig. 5.

After 1 h, the DC% measured for the control groups was not significantly higher in LC than DC (73.3% and 71.7%, respectively, p = 0.104). The DC% for all interposed LC discs ranged from 61.3% to 76.8%, but slightly lower DC% were found when VE and VM thicknesses increased over 1.5 mm (p = 0.001). However, DC discs appeared not consistently affected by the substrate thicknesses, presenting a slightly higher range of DC% (67.2–78.4%) than LC. VE and VM of 1.5 – 2.5 mm thickness were linked with significantly higher DC% for DC resin cement compared to LC cement (p = 0.001).

The DC% of LC discs negatively correlated with VM thickness (r^2 = 0.96, p = 0.001), but not with other materials.

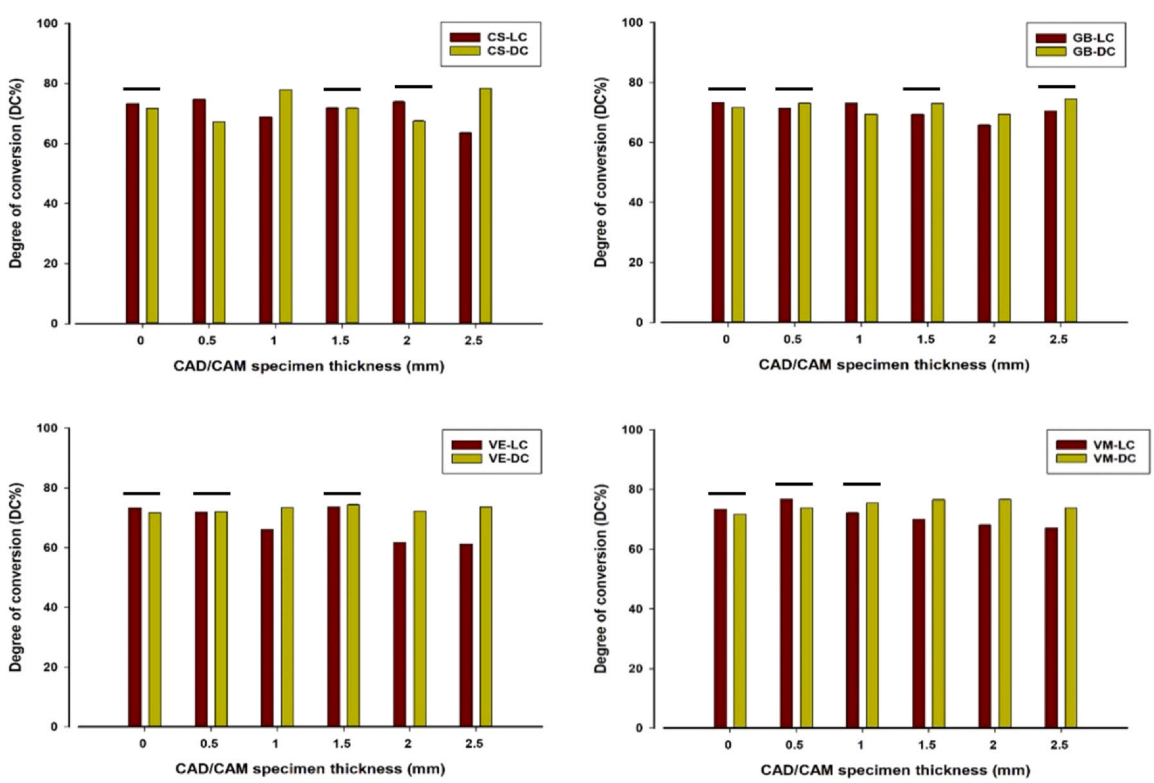

Fig. 5 – Mean DC% for light-cured (LC) and dual-cured (DC) luting cements after 1 h post-photoactivation through each CAD/CAM substrate (CS, GB, VE, and VM). Horizontal lines above the error bars indicate no statistically significant differences (Paired t-test, p > 0.05).

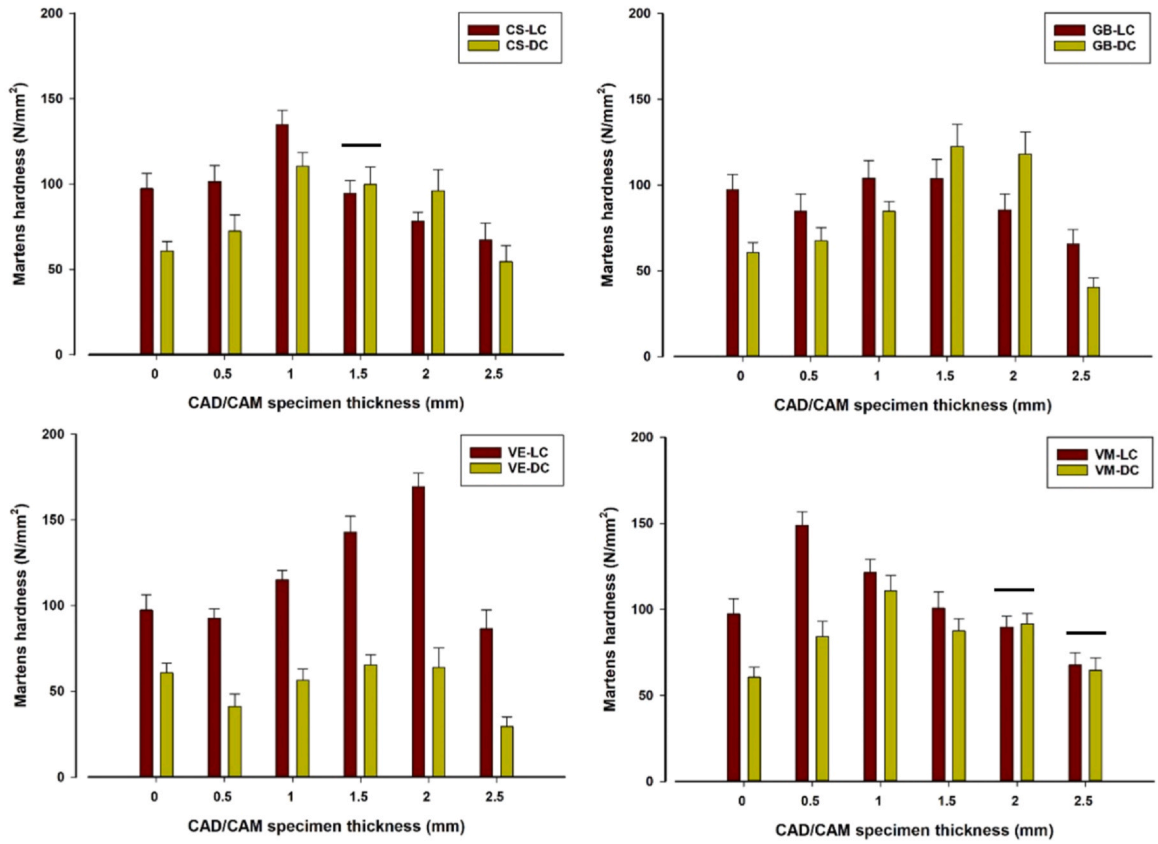

Fig. 6 – Martens hardness (H_M) for light-cured (LC) and dual-cured (DC) luting cements measured at top surface after 1 h post-photoactivation through each CAD/CAM substrate (CS, GB, VE, and VM). Horizontal lines above the error bars indicate no statistically significant differences (Paired t-test, p > 0.05).

3.2.2. *Martens hardness (H_M)*

H_M for luting cements after 1 h post-curing through each CAD/CAM material are presented in Table S4 and graphically illustrated in Fig. 6.

The hardness of the luting discs was significantly affected by the CAD/CAM materials and their thickness ($p = 0.001$). In the control cements, LC were significantly harder than the DC discs (97.3 and 60.6 N/mm^2 respectively, $p = 0.001$). Similarly, overall H_M data of the interposed discs showed that LC were significantly harder than DC (65.5 – 169.3 N/mm^2 and 29.4 – 122.7 N/mm^2 respectively, $p = 0.001$), with few exceptions. The H_M data for the interposed luting discs roughly followed a bell-shaped pattern where hardness was higher underneath mid thickness ranges (1 – 2 mm) but reduced at extremities (0.5 and 2.5 mm).

Curing through 0.5 and 1 mm-thick *resin composites* (CS and GB), resulted in harder LC discs than DC (84.6–135 N/mm^2 and 67.5–110.5 N/mm^2, respectively). At 2.5 mm thickness, although the hardness decreased in both types of luting cements, the dual cured cement was significantly softer than the light cured ($p = 0.001$).

With VE substrates, LC discs were considerably harder than their DC equivalents, regardless of thickness (86.4–169.3 N/mm^2 and 29.4 – 65.4 N/mm^2, respectively). Similarly, in VM substrates of 0.5–1.5 mm thickness, LC discs were significantly harder than DC. However, increasing the VM thickness to 2–2.5-mm, resulted in gradual reduction of H_M with no significant differences between LC and DC ($p = 0.41$ and 0.28, respectively).

For LC discs, there was a minor positive correlation between H_M and T′ % of material substrates ($r^2 = 0.3$, $p = 0.002$) but no significant correlation between H_M and DC% (except for VM). The LC cements cured through feldspathic ceramic (VM) exhibited strong positive correlations between H_M *versus* substrate thickness, irradiance and DC% ($r^2 = 0.982$, 0.987, and 0.961, respectively). Also, the irradiance was linearly correlated with the H_M (LC) in GB substrates, and to a lesser degree in CS ($r^2 = 0.987$, 0.975 and 0.472, respectively, $p = 0.001$). No such correlations between $H_{M,}$ DC%, and substrate thickness were found for the DC discs.

4. Discussion

4.1. *General trends*

This study confirmed the existence of significant optical differences between the four aesthetic CAD/CAM substrates fabricated in five clinically relevant thicknesses ranging from ultra-thin veneers to onlays or crowns (0.5–2.5 mm). Light attenuation parameters: A' and O_p, and reduced translucency, expressed by either TP or T′ % - varied with thickness of these CAD/CAM substrates and were inter-related in consequence of their mathematical definitions.

The overall irradiance of the blue LED used (1200 mW/cm^2 and 24 J/cm^2) was reduced by nearly 32–85% when interposed by increasingly thick CAD/CAM substrates. However, the polymerisation of the interposed Ivocerin™-containing cements, measured after 1 h post-cure, varied considerably in terms of H_M more than DC%. An approximately bell-shaped pattern was observed for the hardness of the luting cements as a function of thickness in all resin-based materials except underlying feldspathic ceramic (VM) where hardness *linearly* correlated with thickness and light irradiance. Overall, LC discs were harder than or equivalent to DC discs underneath VE and VM but this relationship (between LC and DC) was inconsistent underneath CS and GB. Therefore, the first two null hypotheses were rejected, and NH 3 was only partially rejected.

4.2. Light transmission through CAD/CAM aesthetic materials

Unlike clinical reality, the optical measurements were obtained with flat polished specimens without any surface treatment such as etching and air abrasion which usually increases the roughness and light scattering [44]. Also, no bonding agent was applied to the specimens - for standardisation purposes.

Results confirmed the effect of thickness and composition of the substrates on their translucency, in line with previous studies [1,30,45]. At a specific thickness and wavelength (455 nm), the relative ranking of the four materials differed significantly in each optical parameter measured (TP, I_t, and T′ %), with few exceptions. However, the variation in TP between the four materials minimised at increased thicknesses (2–2.5 mm) with only VE being significantly the least translucent. These relatively less favourable optical results for PICN concur with several comparable studies [1,23,30,33,45].

Translucency, in addition to its role in matching the natural aesthetics, regulates the light irradiance and energy available to polymerise the underlying luting cements [41,46]. Within a given restoration thickness, translucency could be expressed using either translucency parameter, wavelength-specific transmittance or contrast ratio [46]. TP and T′% were linearly correlated ($r^2 = 0.967$, $p = 0.0001$) where the standard TP_{Lab} was calculated based on the CIE_{Lab} colorimetry difference of each substrate against black and white standard backgrounds [47]. A better fit of data could be obtained using the CIE 2000 formula for translucency in case of comparing difference thresholds in TP_{00} [48]. However, the wavelength-specific transmittance seems more informative whenever matching absorption peaks of the photoinitiator in the underlying luting cement [38,46]. For example, camphorquinone (CQ), a frequently used photoinitiator, has an absorption range of 400–500 nm (absorption peak around 470 nm) [24]. Whereas, activation of Ivocerin™ can be achieved by a spectrum ranging from 400 to 430 nm (absorption peak around 412 nm) [21] and in this study it peaked around 455 nm.

In general, using a LCU with greater light irradiance, beam homogeneity and required spectral emission should increase the light reaching the luting cements [49]. However, the DC% of Variolink Esthetic did not significantly increase when using different light sources (LED *versus* QTH) [41] or a polywave *versus* single-peak LCU [50]. Recent studies found that Ivocerin™ was sensitive to even an extended wavelength range from 360 up to 460 nm [38,50,51]. Unlike CQ, Ivocerin™ can breakdown directly into free radicals with relatively fewer photons and without additional co-initiators such as tertiary amines [21]. This has the advantage of using almost any LED-LCU for photoactivation [51] and requiring comparatively shorter exposure times [50,52]. Therefore, a standard 'blue' LED curing unit (430–480 nm) emitting 1200 mW/cm^2 with a power of about 700 mW should be adequate for polymerising Ivocerin™- containing resin cements.

In the present study, the incident irradiance was 1200 mW/cm^2 but the transmitted irradiance ranged from 181.9 to 819 mW/cm^2, depending on substrate thickness. A similar study recorded a drop in irradiance from 700 mW/cm^2 to 100 mW/cm^2 with an interposed 2-mm resin composite substrate [18]. Concurring with previous studies, linear inverse correlations were confirmed for substrate translucency [23,32,33,40] and light irradiance [30,34,45] *versus* thickness. The minimum irradiance range to achieve efficient resin polymerisation is 400–500 mW/cm^2 for 20–40 s with a 1 mm thickness substrate, depending on photoinitiator type and content [24,32]. Our CAD/CAM substrates of 0.5- and 1-mm thickness transmitted sufficiently high irradiance above 500 mW/cm^2 in the increasing order of VM = GB > CS > VE. However, further increasing substrate thicknesses to 1.5–2.5 mm, significantly reduced irradiance (182–406 mW/cm^2) denoting an irradiance loss of 66–85%. This corresponds to a previous study where irradiance decreased by 60–95% through various aesthetic restoratives [30].

4.3. Polymerisation of the luting cements (1 h post-curing)

Polymerisation of resin cements typically progresses over 24–48 h before complete maturation [15,25,53]. Unfortunately, poor marginal adaptation of the restoration exposing the luting cement to the oral environment could compromise its integrity over time [25]. This study, however, measured cement polymerisation after 1 h - simulating a clinical session where an *early adequate* polymerisation is critical to withstand any additional mechanically-stressful clinical procedures. The long-term consequences associated with poor cement polymerisation may begin with increased water absorption [54,55], leading to discoloration [56], release of residual monomers, causing sensitivity or toxicity [2], mechanical degradation [57], compromised bond strength and eventually restoration loss [27,58].

DC% indicates the overall conversion of the C═C group but does not completely reflect the integrity of the cross-linked polymer [29]. In general, DC% for the resin-based materials range from 55% to 77% [20,59]. However, there is no consensus on the minimum clinically acceptable DC% for luting cements. Our results showed that DC% for the interposed LC and DC discs (61–78%) were within the ranges measured immediately after light curing - in line with previous studies [7,20,25,58].

Thickness variation of VE substrates had no effect on the DC% of the underlying DC cements (72.1–74.4%). A similar observation was recorded in a comparable study for DC cements cured underneath 1–2 mm-thick VE and resin composite substrates [23]. Our results revealed relative stability in DC% of the Ivocerin™-containing cements cured through increasing thicknesses, as seen with other luting cements [24,49,60].

However, Martens hardness data showed significant differences between the luting cements in terms of curing mode and substrate optical features and thicknesses. H_M of any luting resin cement is influenced by its composition [61], the setting parameters applied during the force-controlled indentation [62] and timing of the measurement [25]. Therefore, H_M data for each cement were compared to its control within the study variables. Nevertheless, the ranking and trends of the studied materials were comparable to previous studies [25,63].

In the control cement discs, LC were significantly harder than DC discs. The same pattern was observed in the interposed luting discs, except for discs underneath the resin composites (CS and GB). However, other studies reported the opposite trend - with harder DC than LC cements when photoactivated through different ceramic substrates [33,41]. The timing of the hardness measurement could explain this difference where hardness can develop towards polymerisation maturation after 24 h up to 7 d *versus* 1 h in this study. A study revealed that after 24 h, no significant differences were found between Knoop hardness of luting cements cured underneath 2 and 4 mm-thick ceramic substrates [15]. However, this does not necessarily mean that the polymerisation will be enhanced by the chemical activation of the DC cement alone [24,49,60]. A study revealed no significant improvement in the hardness of DC cements after 24 h of photoactivation through 3 mm thick ceramics [49]. With limited light transmission due to increased ceramic thickness, the cement hardness reduced by nearly 60–70% regardless of the cement curing mechanism [60]. These low hardness results were found, in other studies [17,49], comparable to DC resin cements applied without any photoactivation. However, our DC% data on the DC Ivocerin™-containing cements was boosted by almost *any* minimal amount of light energy received (2.08–7.92 J/cm^2). Further monitoring of the hardness development of these cements, receiving variable light energy, is required.

Overall, H_M data followed a bell-shaped pattern where the greater hardnesses were recorded roughly around mid-range thickness (1 – 2 mm) and the lowest at 0.5- and 2.5-mm thickness. With 2.5 mm thick substrates, H_M for DC discs reduced to a minimum of 6.8% (VM) to a maximum of 51% (VE), compared to their control. Luting discs underlying each thickness of VE substrates exhibited divergences between the two curing mechanisms, compared to other materials (DC cement was distinctively softer than LC, $p = 0.001$). This is consistent with the reduced translucency of VE, as previously discussed. However, CS and GB resin composites bonded with DC cement showed inconsistent H_M results, similar to previous observations [23,33,40].

Feldspathic ceramic (VM) was the only substrate material showing strong linear correlations between DC% and H_M *versus* their translucency to LC cements. Similarly, two recent studies found a strong correlation between H_M and DC% in LC cements measured after 24 h, either interposed with 1 mm-thick ceramic or not [25,63]. However, the difference in hardness between LC and DC cements was not significant with VM substrates at 2 and 2.5 mm thickness. Therefore, the results suggest that LC and DC could be used indifferently for bonding ceramics (2–2.5 mm thickness), though more studies are required to verify this.

Unlike restorative resin composites, aesthetic resin cements are designed to be cured through substrates of different translucent materials of a certain thickness. Results showed that passing excessive light irradiance to the luting cement discs cured directly (control; 1200 mW/cm^2) or through 0.5 mm-substrate (748 mW/cm^2), negatively affected their hardness development. This phenomenon, also noted in previous studies [23,26,49], might be related to the type and concentration of the photoinitiators extant in the resin cement where overexposure might disturb their activation mechanism causing a higher free radical termination rate [64], which could compromise their polymerisation and subsequent hardness development.

In contrast, other than the light irradiance, the necessity of increasing the exposure time to ensure receiving sufficient radiant exposure to activate the photoinitiator has been discussed for substrates having greater light-attenuating qualities [12,45] such as increased thickness, darker shades [1] and opacity [23,65]. Following recommendations from previous research [23,64], the exposure time for photoactivating the DC luting cement specimens was made *double the manufacturer's guide* to ensure adequate early C=C bond conversion. However, considering their H_M results, extending the exposure time did not compensate for inadequate light transmission to DC discs cured through all VE substrates. This finding matches other studies in which neither implementing longer curing time nor higher irradiance light improved the polymerisation results of luting cements cured through substrates with increased light-attenuating features [15,16,18].

Light transmission (I_t) through increased thicknesses of CAD/CAM monolithic aesthetic materials varied considerably, thereby influencing the polymerisation (H_M) of each underlying luting cement. However, as observed elsewhere [7,23], DC% data were less sensitive in reflecting the influence of substrate-dependent variables such as thickness and translucency. Combining H_M results from all material-thickness substrates suggest that LC is a better choice than DC, in terms of early hardness development (1 h). This choice becomes more appealing if greater discolouration resistance is required [6,10]. Nevertheless, more research is required to verify the hardness development of LC and DC after complete polymerisation.

5. Conclusions

Interposing increasing thicknesses of CAD/CAM aesthetic restorations, from 1.5 to 2.5 mm, significantly reduced the blue-light transmission for polymerising underlying light-and dual-cured, Ivocerin™-containing resin cements. However, the cement hardness after 1 h post-photoactivation differed considerably across the four substrate materials. Light-cured cement underlying feldspathic ceramics and polymer-infiltrated ceramics achieved greater early hardness than the dual-cured type regardless of substrate thickness. Equivalent outcomes (LC > DC) were only observed in resin composites of 0.5–1.0 mm thickness. In contrast, the degree of conversion of resin cements was relatively stable under the study conditions with the dual-cured type exhibiting slightly greater degrees of conversion.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.dental.2022.11.016.

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